[O Interim Technical Report, For 25 Sept to 25 Dec. 1962]

EVALUATION OF REGENERATIVE FUEL CELL

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Prepared by

J. J. Rowlette J.
Project Supervisor

PRICES SUBJECT TO CIRCLES

Approved by

B. M. Wilner, Manager Chemical and Fluid Systems Department Approved by

J. Newstern

J. Neustein, Manager ADVANCED POWER SYSTEMS DIVISION

084846/
ELECTRO-OPTICAL SYSTEMS, INC., PASADENA, CALIFORNIA

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1. INTRODUCTION

Most of the original objectives of the first phase of the present program have been met. The objectives were: (1) to study the parameters affecting the performance of hydrogen-oxygen fuel cells, (2) to study doped silver oxide electrodes to increase their capacity on the first silver plateau, (3) to study the feasibility of hydrogen storage within a palladium electrode for possible use in a hydrogen-silver oxide hybrid fuel cell, and (4) to study certain radioactive doped electrodes for use as oxygen electrodes.

The individual effects on hydrogen and oxygen electrodes have been determined for pressure, temperature, and electrolyte purity, and the effects of mixed platinum-palladium catalysts have been determined for the hydrogen electrode. In the latter experiments it was shown that mixed catalysts are better than either catalyst alone, especially at higher current densities.

Silver oxide and ten different doped silver oxides were tested for electrical resistivity. Manganese, and especially cobalt, showed a significant lowering of the resistivity of silver oxide. These two doped oxides were then compared with the undoped oxide and it was shown that with cobalt a small but definite improvement over pure silver oxide can be realized. Silver oxide electrode capacities as high as 77 percent of theoretical have been achieved on the first plateau.

The feasibility of hydrogen storage within a palladium electrode was demonstrated. Even when charged at 155 ma/cm² the hydrogen is absorbed into the electrode almost as fast as it is formed, and 67 percent can be recovered electrochemically when discharged at that rate. Close to the theoretical amount of hydrogen was absorbed, but it was

not shown whether the limitation here was due to the palladium or to the capacity of the silver oxide electrode.

There was no definite correlation shown between the radioactive doped silver electrodes and the control. The presently used platinum catalyzed porous nickel electrodes have less polarization than any of the silver electrodes and for that reason no further work with the radioactive electrodes is anticipated.

Phase II of the program, concerned with single cell development, is under way. The initial studies are concerned with increasing the amount of stored electrolyte. A cell has been designed and constructed which has a major amount of stored electrolyte behind the electrodes. Initial results with the cell have been successful.

2. HYDROGEN AND OXYGEN ELECTRODE STUDIES

The effects of several factors which might influence electrode performance of hydrogen-oxygen fuel cells have been studied. All hydrogen electrodes and all oxygen electrodes except those for the radioactive studies were porous nickel catalyzed with platinum (or a platinum-palladium mixture in most cases) as described in a previous report (Ref. 1).

The method of measuring individual electrode polarizations was also described in the above-mentioned report. It should be born in mind that these measurements include ohmic resistance. For all hydrogen electrodes reported in this section, the polarizations refer to the potential of the working electrode minus the potential of an exactly similar electrode in the same solution. For the oxygen electrodes, however, the irreversible nature of that electrode precludes its use as a reference electrode and in all cases a reference electrode of $Ag_{2}0$ was used. But the values reported in this section for the oxygen electrode do not represent the oxygen electrode potential minus the Ag₂0 potential. The hypothetical reversible oxygen electrode potential is 1.229 volts at 25° and the reversible $\mathrm{Ag}_2^{}$ 0-Ag potential is 1.172volts (Ref. 2). The potentials of Ag_20 vs. H_2 and O_2 vs. H_2 are pH independent except at very high electrolyte concentrations where the activity of the solvent deviates significantly from unity. The hypothetical reversible potential of an actual cell will also be a weak function of the gas pressures and the temperature, but to a good approximation, under the conditions prevailing in these experiments, the reversible oxygen potential (hypothetical) will be about 0.06 volt higher than that for ${\rm Ag}_2^{}{\rm O}_{\cdot}$ It becomes convenient then to subtract this amount from the measured value of the $\mathbf{0}_{2}$ (working) electrode minus the $\mathrm{Ag}_2\mathrm{O}$ (reference) electrode and give plots which to a close approximation represent the extent of polarization of the oxygen electrodes. The results presented in this way are consistent with the hydrogen electrode polarization, i.e., the sum of the two electrode polarizations plus the cell potential is invariably 1.23 volts to within the accuracy of the measurements.

2.1 Pressure Dependence

Pressures were varied from 50 to 95 psig in these experiments. In this range, the performance of the hydrogen electrode is independent of gas pressure. The oxygen electrode, however, varies significantly within this range. The results are shown in Fig. 1. The oxygen electrode was porous nickel with 15 mg Pt/cm².

In addition to the effect of total pressure, it has also been found that oxygen electrodes are more sensitive to flooding than are hydrogen electrodes. If the gas pressure at either side is increased over that at the other, then the electrode at the low, pressure side begins to show severe concentration polarization. The hydrogen electrode can tolerate a differential of 1 to 2 psi while the oxygen electrode cannot. In fact, the oxygen electrode often works best if the oxygen pressure is about 1 psi higher than the hydrogen pressure. These results are presumably due to flooding and the difference in behavior of the two electrodes likely reflects the lower diffusion rate through the electrolyte film for oxygen.

2.2 Temperature Dependence

Polarization studies were made at 28°, 49° and 68°C. The results are given in Fig. 2. The temperature measurements were made with a chromel-alumel thermocouple whose junction was at the center of the fuel cell and about a millimeter behind the oxygen electrode. The oxygen electrode was similar to the one described in Section 2.1 and the hydrogen electrode was a mixed catalyst electrode as described in Section 2.3. The mixture was 50 percent Pt, 50 percent Pd with a total of 10 mg/cm² of metallic catalyst.

It will be noted that the room temperature polarization of the hydrogen electrode is somewhat greater than that for the corresponding

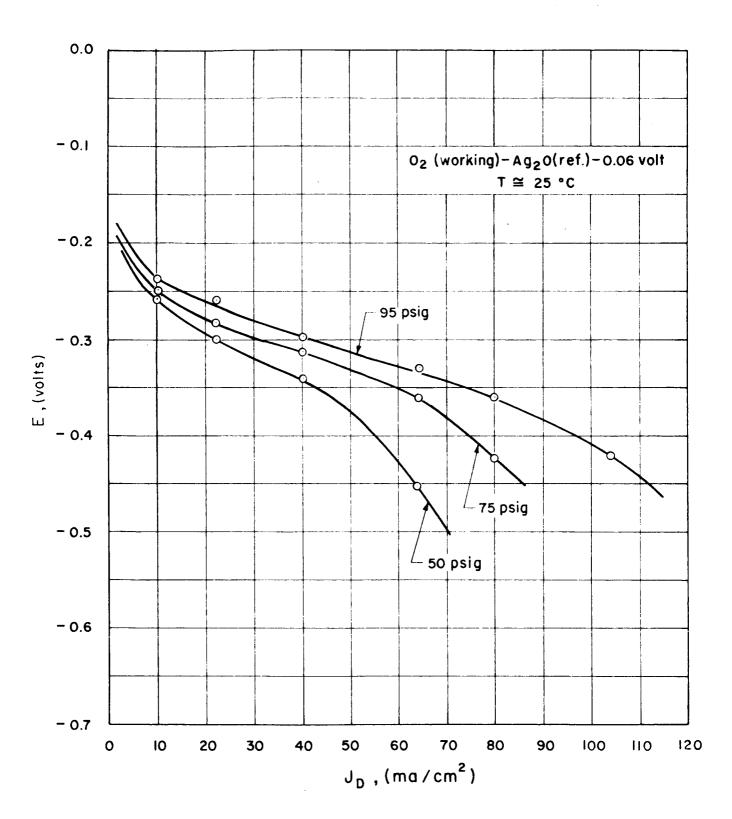


FIG. 1 PRESSURE DEPENDENCE OF AN OXYGEN ELECTRODE

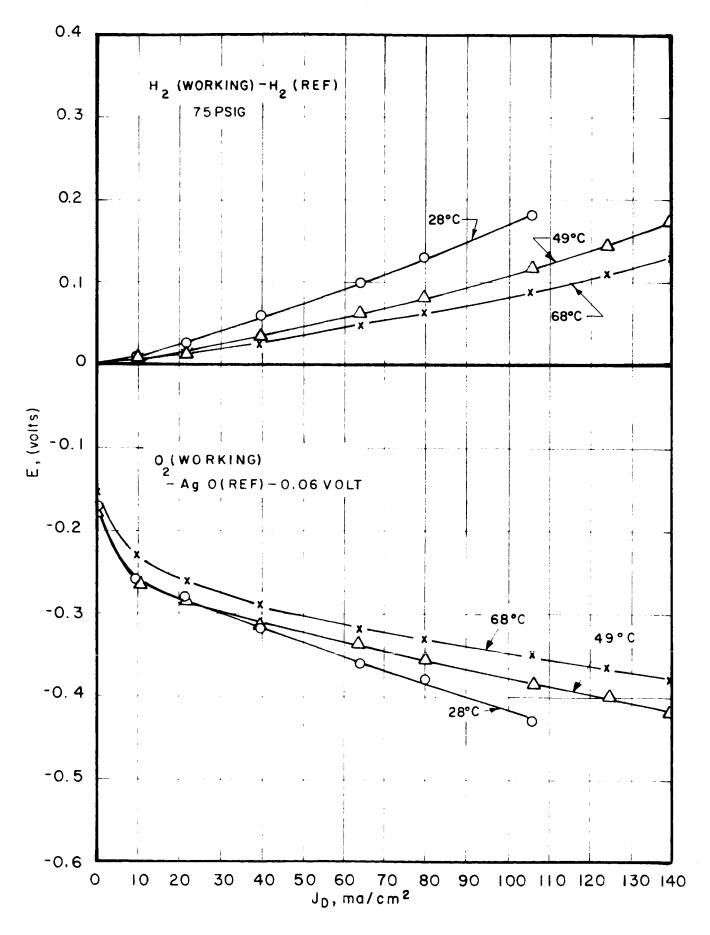


FIG. 2 TEMPERATURE DEPENDENCE OF POLARIZATION

electrode reported in Section 2.3. Some of the electrodes were used in a great many experiments and it has been observed that these electrodes deteriorate slowly with handling, i.e., being taken in and out of a cell. All results shown in this report are for series of experiments run within as short a period as possible so that all variables could be controlled as accurately as possible. Reproduceability is usually good between identical experiments run close together in time.

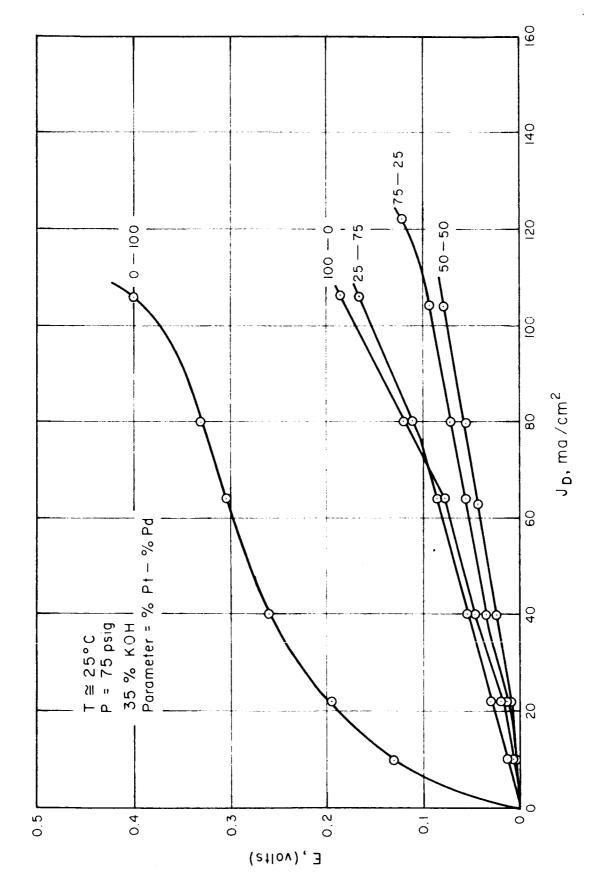
Although these electrodes deteriorate gradually with handling, there is good evidence that thay are stable with time, if they remain within the cell.

2.3 Effect of Mixed Catalysts

For hydrogen electrodes it has been discovered that mixed platinum-palladium catalysts are superior to either catalyst alone. A study was made in which the ratio of platinum to palladium was varied systematically. The results of the electrode polarization characteristics are shown in Fig. 3 for the discharge process and in Fig. 4 for the charge process. The pressures were varied between 50 and 95 psig but the curves in all cases were essentially identical to the ones shown here.

It is apparent that the optimum ratio occurs at about 50 percent platinum and 50 percent palladium, but no explanation for this observation can be advanced at this time. There was, however, one fact noted in connection with electrodes plated with the mixed catalysts. They are visibly blacker than electrodes with only one catalyst and their enhanced activity may be associated with the manner in which the activating metal plates out. Thus, the coplating of palladium along with platinum might result in a finer particle size.

There has not yet been a corresponding series of experiments made with oxygen electrodes, but preliminary observations on overall cell potential (made in a cell without reference electrodes) would indicate that the effect here is minimal.



. 3 HYDROGEN ELECTRODE POLARIZATION, DISCHARGE

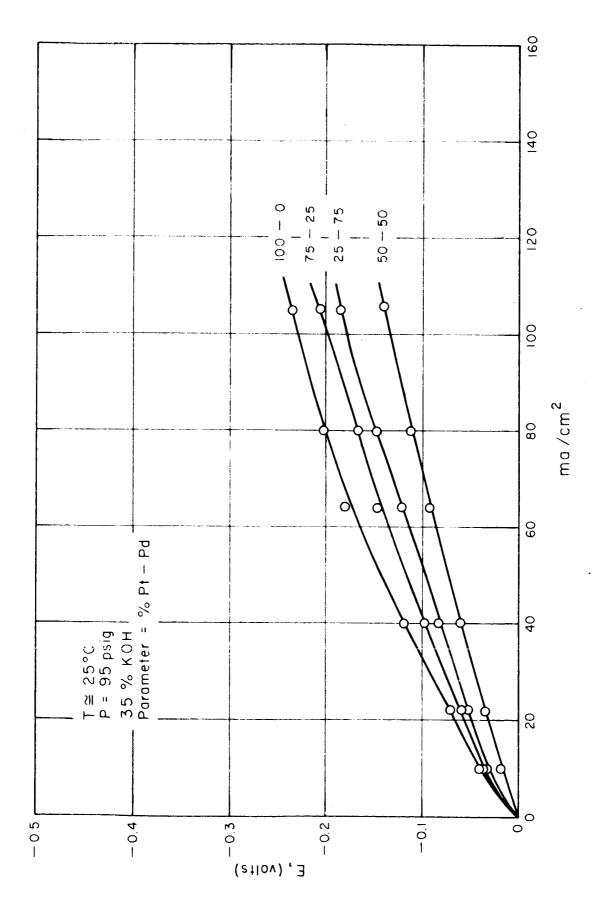


FIG. 4 HYDROGEN ELECTRODE POLARIZATION, CHARGE

The plating itself is accomplished by the method described previously (Ref. 1), but the plating solution is a mixture of ${\rm H_2PtCl}_6$ and ${\rm PdCl}_2$. The amount of metallic catalyst totaled 10 mg/cm 2 in every case except for the electrode with palladium alone, which had 7.7 mg/cm 2 .

2.4 Electrolyte Purity

The reagent grade KOH used in these experiments contains enough carbonate that a 35 percent solution made from water free from $\rm CO_2$ has a $\rm CO_3^=$ concentration of about 0.032 M. Cells with the least polarization have been obtained using an electrolyte free from carbonate. In order to remove the carbonate ion from the electrolyte, $\rm Ba(OII)_2$ is added, which precipitates carbonate as $\rm BaCO_3$. This is allowed to settle and the solution is used from the top. In order to ascertain the effect of an excess of either $\rm Ba^{++}$ or $\rm CO_3^=$, experiments were performed in which various known concentrations of $\rm Ba^{++}$ or $\rm CO_3^=$ were present. The results are shown in Fig. 5.

The experiments were run several times, and although the curves of Fig. 5 are the most nearly typical of any set of experiments, factors other than electrolyte purity influence reproduceability with the result that only a few real conclusions can be drawn at this time. The following conclusions for the discharge process can be drawn:

- 1. High concentrations of carbonate ion always cause a considerable increase in polarization at the hydrogen electrode.
- 2. For the hydrogen electrode there is no consistent difference between 0.29 M Ba $^{++}$, 0.032 M Ba $^{++}$, and 0.032 M C0 $_3^{=}$, but the "pure" electrolyte is generally a little better.
- 3. For the oxygen electrode 0.64 M $\rm C0_3^=$ always causes the greatest increase, followed by 0.29 M $\rm Ba^{1+1}$.
- 4. In the current density range of about 60 to 100 ma/cm^2 , the oxygen electrode consistently operates best with a slight excess of barium ion, and the slope of the curve in this region is unexplainably small.

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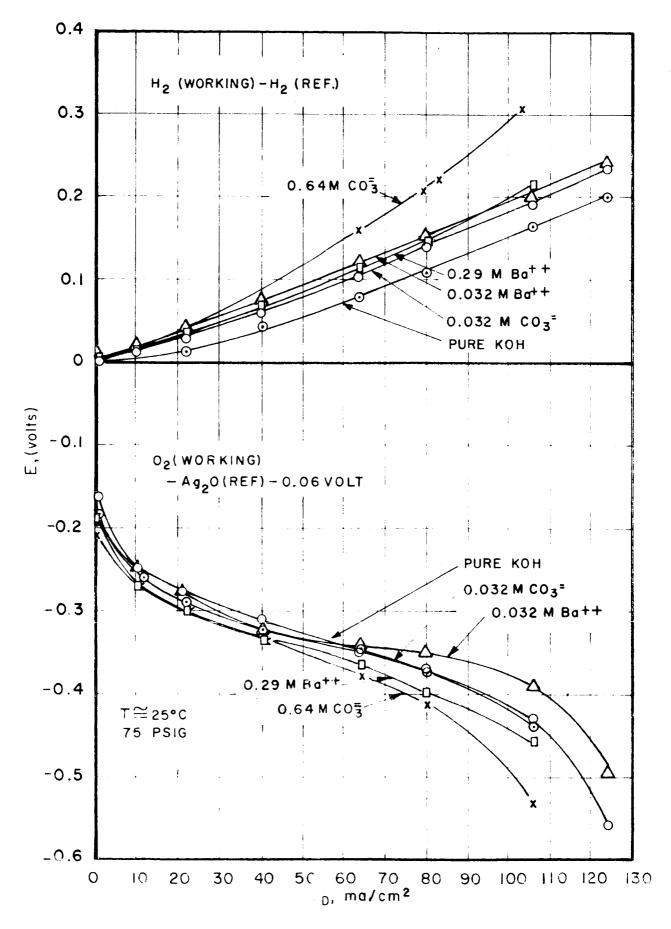


FIG. 5 EFFECT OF ELECTROLYTE PURITY ON ELECTRODE POLARIZATION

2.5 Radioactive Electrodes

Four sintered silver electrodes, made from 100--200 mesh silver powder and containing a silver screen for strength, were made up for evaluating radioactive electrodes for oxygen. Three were sent to Yardney Electric Company where two of them were doped with Po^{210} , an a emitter. The third was doped with Pm^{147} , a β emitter. The fourth electrode was saved for a control. All four were tested as oxygen electrodes in a hydrogen-oxygen cell at room temperature and at 70°C . No definite correlation between radioactivity and electrode polarization was demonstrated. The results are shown in Figs. 6 and 7, together with one of our presently used platinum blacked electrodes for comparison.

In Figs. 6 and 7 the electrode designations are as follows:

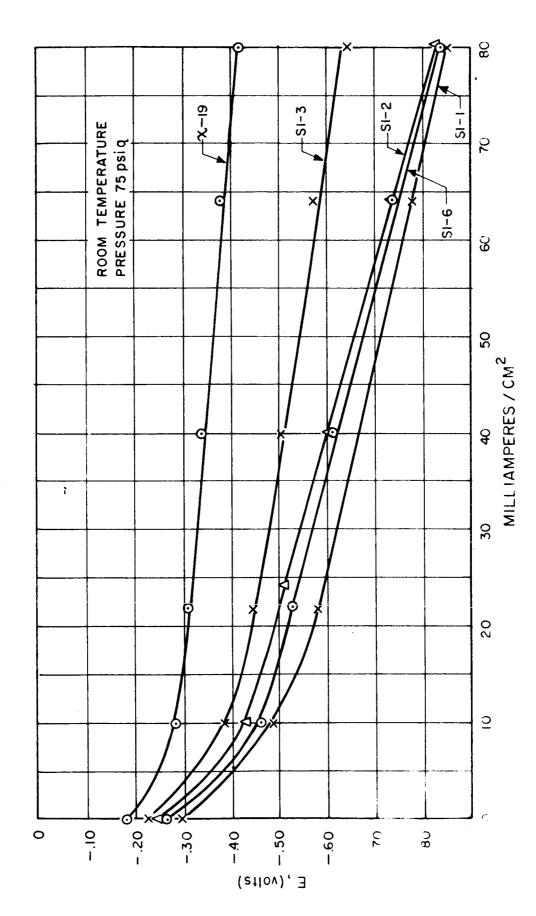
SI-1 Sintered silver, doped with Pm 147

SI-2 Sintered silver, doped with Po 210

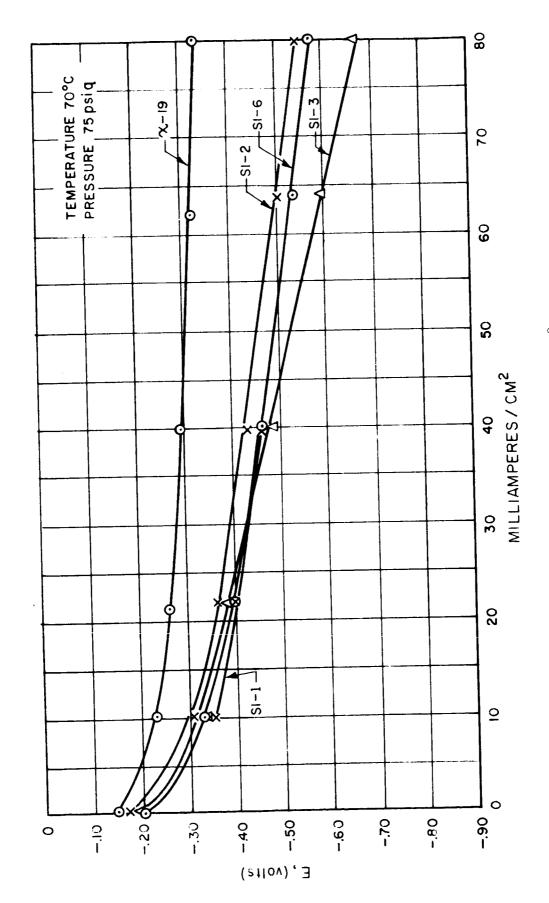
SI-3 Sintered silver, doped with Po 210

SI-6 Sintered silver, undoped

X-19 Porous nickel, platinum catalyzed (15 mg/cm 2)



POLARIZATION OF RADIOACTIVE DOPED ELECTRODES, ROOM TEMPERATURE FIG. 6



.G. / POLARIZATION OF RADIOACTIVE DOPED ELECTRODES, 70°C

3. ELECTROLYTE STORAGE EXPERIMENTS

A new cell has been designed and constructed for capacity experiments. An analysis of the energy storage/weight capabilities of the hydrogen-oxygen system quickly reveals that this ratio will be limited more by the amount of stored electrolyte than by the weight of the gas storage cylinders. In all cells studied to date in this laboratory, the only electrolyte storage has been between the electrodes. The new cell, shown in Figure 8. is designed to allow electrolyte storage behind the electrodes. The gas for each electrode is brought up through a hole in the electrolyte storage bed to a screen immediately behind the electrode. The gas port sleeves serve a triple function. They allow gas passage up to the nickel screen, where the gas is then dispersed across the entire back of the electrode, they make electrical contact to the electrode, and they determine the spacing (at least at two points) between the electrodes.

The storage behind the hydrogen electrode was made twice as much as that behind the oxygen electrode because a consideration of the individual electrode reactions shows that twice as much water is consumed or produced (for the charge or discharge process, respectively) at the hydrogen electrode as at the oxygen electrode. These electrode reactions are

$$\text{H}_2 + 2 \text{ OH} \xrightarrow{\text{discharge}} 2 \text{H}_2 \text{O} + 2 \text{e}^{-1}$$

and
$$2e^{-} + \frac{1}{2} 0_{2} + H_{2}0 \xrightarrow{\text{discharge}} 2 \text{ OH}^{-}$$

3310-Q-2

The first experiment with this new cell is now in progress. A 30-hour charge at 20 ma/cm 2 has been completed and the cell is now being discharged at the same current density. During the course of the 30-hour charge, the cell voltage rose gradually from 1.62 to 1.73.

DAG BORT SLEEVES

3310-Q-2

FIG. 8

EXPERIMENTS

HYDROGEN-OXYGEN FUEL CELL FOR ELECTROLYTE STORAGE

4. SILVER OXIDE ELECTRODE STUDIES

In order to improve the polarization characteristics and capacity on the first charging plateau of a silver oxide electrode, experiments were conducted on the effect of doping the silver oxide. There is evidence that the capacity on the first plateau is limited by the high resistivity of Ag₂0. Thus, a prevalent theory to account for the fact that much less than the theoretical charge is accepted on the first plateau is as follows: oxidation of Ag to Ag₂9 occurs only on the surface, and when the surface of the electrode becomes completely covered by the high resistance film of Ag_20 , then Ag_20 is oxidized to AgO and the potential of the electrode increases to that of the second plateau. Essentially all of the electrode can now be oxidized since the Ag0 formed can thermally oxidize Ag to Ag_20 and itself be reduced to $\mathrm{Ag}_2\mathrm{O}$. The resistivity of $\mathrm{Ag}\mathrm{O}$ is reported to be from seven to nine orders of magnitude less than that of ${\rm Ag}_2{\rm O}$, or approximately 0.01 to 10 ohm-cm (Ref. 2). That the high resistivity of ${\rm Ag}_2{\rm 0}$ is responsible for the limited capacity on the first plateau, and not some other factor such as availability of electrolyte, is demonstrated by some observations by T. P. Dirkse (Ref. 3). It has been our observation, as well as that of Dirkse, that the electrochemical action occurs outward from the grid or, in the case of most of our cells, from the contact between the electrode and the body of the cell at the back of the electrode. On the oxidation of $\mathrm{Ag}_2\mathrm{O}$ to AgO , Dirkse has this to say, "Thus, the resistance of the $\mathrm{Ag}_2\mathrm{O}$ seemed to make it easier for AgO to form at the grid where the amount of electrolyte was small rather than at the electrode surface where there was plenty of electrolyte".

A program was conducted in which ten samples of Ag_20 were each doped with 0.5 atom percent of a dipositive element. Their resistivities were determined and compared with that of undoped Ag_20 . Then two of the best of these were studied as silver oxide electrodes and again compared with the undoped Ag_20 .

4.1 Resistivity of Doped Silver Oxide

Silver oxide was doped by a coprecipitation method. To a neutral solution of ${\rm AgNO}_3$ and the nitrate of the impurity metal, a 35 percent KOH solution was added while the whole solution was being stirred with a magnetic stirrer. The solid phase solubilities of the oxides in question are in general not known, but the amount of impurity oxide formed would give 0.5 percent on an atomic basis on the assumption that its solubility in ${\rm Ag}_2{\rm O}$ is that great.

Table I shows resistivity values at room temperature for all samples, including the undoped Ag_20 . Values at $100^{\circ}C$ are also given for the doped oxides except the cobalt doped oxide, where the resistivity was too low for the measuring apparatus and the cadmium and mercury doped oxides, where the samples were broken. The room temperature values for the latter two are therefore uncertain also. The samples for Table I were prepared by placing the slightly wet oxides in a rubber cylinder about 1 cm in diameter and enerting a hydrostatic pressure of approximately 250 psi. They were then dried overnight at $100^{\circ}C$ and placed in a dessicator until they were ready for use. Each sample was approximately 2 rm thick but there was some variation from one to another. The resistivity of Ag_20 has been reported elsewhere to be of the order of 10^{8} ohn-cm. (Ref. 2).

4.2 Polarization and Capacity of Silver Oxide Electrodes

Charging curves on the first silver plateau are shown in Fig. 9 for several sintered silver electrodes. The compositions of these electrodes are as follows:

- S-2-2 Spherical silver (100-200 mesh) + asbestos
- S-3 Raney silver

TABLE I RESISTIVITIES OF DOPED AND UNDOPED ${\rm Ag}_2{\rm O}$

Material	R,25°C	ρ,25°C	R,100°C	ρ,100°C
Ag ₂ 0	1.30	5.20		
Ag ₂ 0 +0.005 Zn0	3.30	15.0	0.03	0.14
Ag ₂ 0 +0.005 Cd0*	(4.90)	(20.0)		
AS20 +0.005 HgO*	(0.56)	(2.25)		
A ₃₂ 0 +0.005 Cu0	1.46	6.30	0.92	0.09
Ag ₂ 0 +0.005 Pb0	1.05	5.50	0.01	0.05
Ag ₂ 0 +0.005 Pd0	0.70	4.40	< 0.01	< 0.06
Ag ₂ 0 +0.005 Mn0	0.10	0.52	< 0.01	< 0.36
$Ag_2^0 + 0.005 \text{ Fe}0$	0.81	4.50	0.02	0.11
Ag ₂ 0 +0.005 Co0	0.02	0.08		
Ag ₂ 0 +0.005 Ni0	0.79	3.95	< 0.01	< 0.05

^{*} Samples were cracked

 $R = resistance, 10^{3} ohms$

 $[\]rho = resistivity, 10^8$ ohm-cm

- S-4 Silpowder No. 130
- S-6 75 percent silpowder, 25 percent carbon

Each of the above electrodes contained a silver screen which weighed about 1.2 gm. This solid silver screen only partially enters into the reaction, with the result that there is some uncertainty in the reactivity of the material under test. For that reason, all other silver oxide electrodes were made without the screen.

If the oxidation of the screen be assumed negligible, then in ma-hrs/gm, the capacity of the S-3 electrode would be 3.28 times as great as shown in Fig. 9 and it would therefore approach the values for those shown in Figs. 10 through 13.

In Fig. 10, are shown the capacities and polarization characteristics of an undoped silver oxide electrode. The electrode was not analyzed for silver content; however, electrodes of this type usually have about 1.1 to 1.3 gm of silver.

Cobalt doped silver oxide electrodes were compared with undoped silver oxide. The results are shown in Figs. 11 and 12. For experiment, the cells were discharged completely through a 1 ohm resistor and charged until the second silver plateau was reached. The silver electrodes were later analyzed, using the Volhard method, to determine the total amount of silver present.

Since the Ag₂0 paste is usually given a final wash with the electrolyte in order to prevent pH gradients, the effect of pH within the electrode was also examined and is shown in Figs. 11 and 12. Finally, in Fig. 13, the results of a charge at 2.5 ma/cm² are shown and compared with those at 40 and 64 ma/cm². The electrode material again was cobalt doped. Some experiments with manganese doped silver oxide were performed but they seemed to offer no improvement over the undoped oxide.

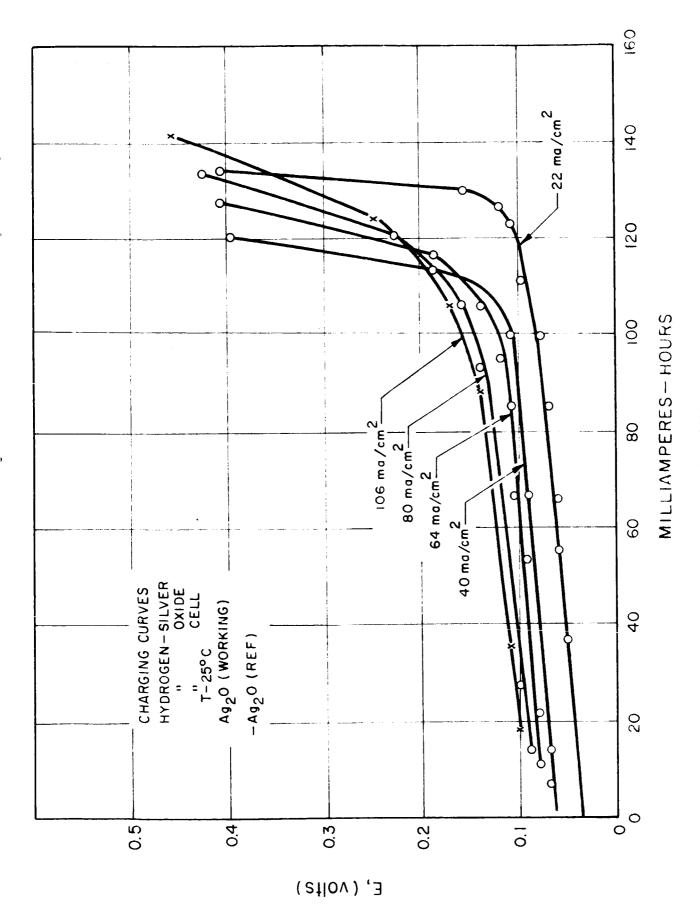


FIG. 9 CHARGING CURVES HYDROGEN-SILVER OXIDE CELL

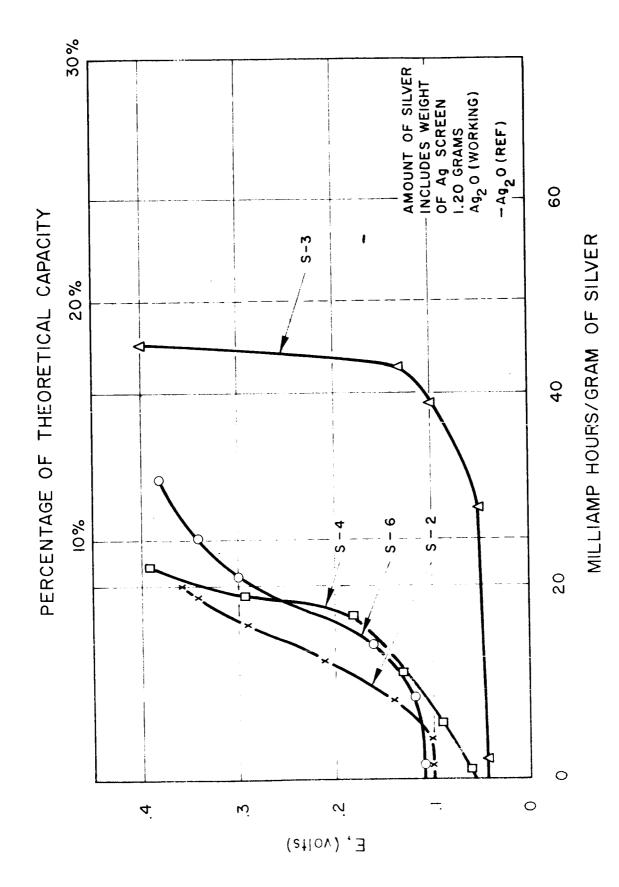


FIG. 10 SILVER ELECTRODE CHARGING CURVES (S - SERIES)

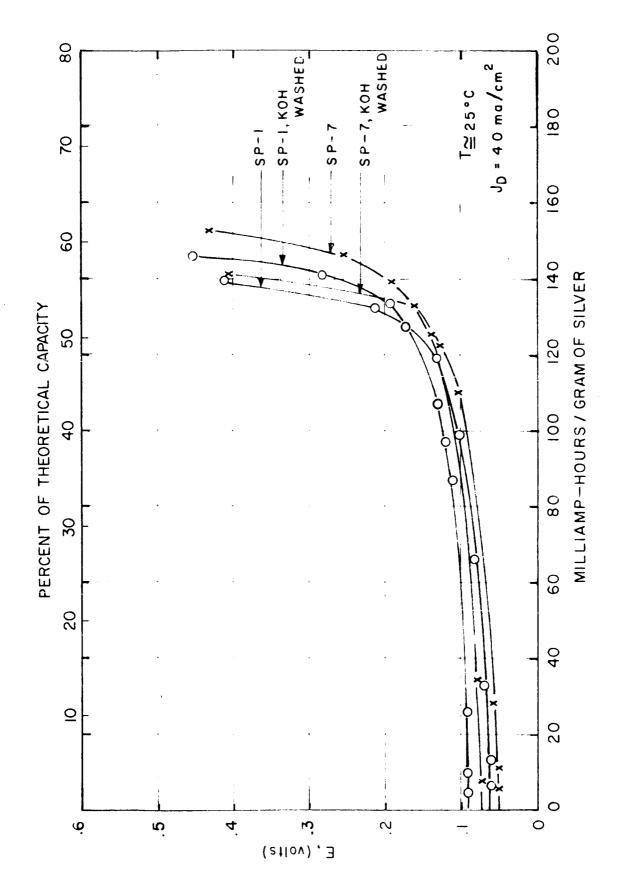


FIG. 11 SILVER ELECTRODE CHARGING CURVES

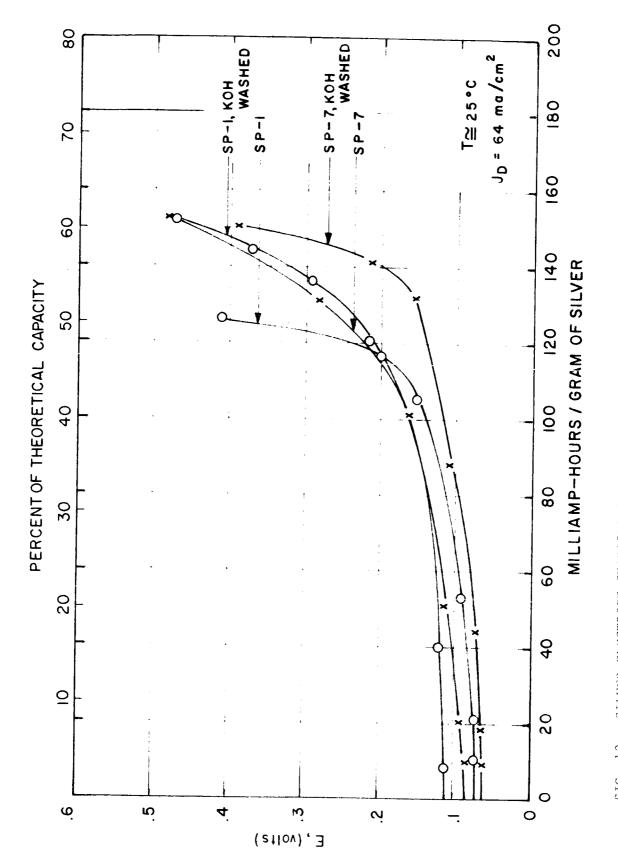
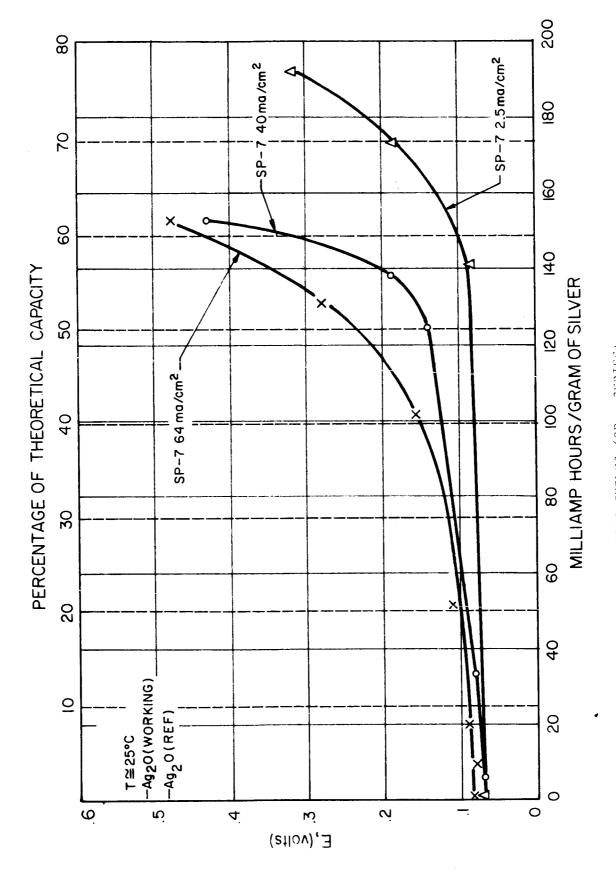


FIG. 12 SILVER ELECTRODE CHARGING CURVES



G. 13 SILVER ELECTRODE CHARGING CURVES (SP - SERIES)

5. HYDROGEN STORAGE IN PALLADIUM

A hydrogen-silver oxide fuel cell would be especially attractive if the hydrogen were stored within the electrode. A hybrid fuel cell would then be obtained, since the hydrogen electrode would be non-consumable, and in this case, there would be no gas pressure storage.

A hydrogen electrode was fabricated by sintering palladium powder onto a fine platinum screen. A palladium black catalyst was applied by thermally decomposing $PdCl_2$ at $500^{\circ}C$ on the sintered palladium electrode. About 7.5 mg Pd/cm^2 was applied in this manner. Pressure was monitored continuously while a hydrogen-silver oxide cell was charged along the first silver plateau, using the sintered palladium for the hydrogen electrode. The object was to determine polarization characteristics and the rate of pressure build-up within the cell.

The results, shown in Figure 14, were generally very satisfactory. It can be seen that there was a very slight increase with time for the three highest charging rates, the gage pressure was always negative except for the 155 ma/cm² rate. At the end of the latter charge there was a positive gage pressure of about 3 psi. The reason for the negative pressures is that the hydrogen side of the cell was swept with hydrogen, in the usual manner, before each run and this was then quickly absorbed by the palladium.

The cells were discharged at the same rates at which they had previously been charged. The amp-hour ratio at 40 ma/cm² was 95 percent; at 80 it was 87 percent; at 124 it was 75 percent; and at 155 ma/cm² the ratio was 67 percent. The number of ma-hours obtained on discharge was 180, 226, 258, and 104 respectively. The weight of palladium (minus the platinum screen) was 1.399 gm. However, it was not shown

that these figures represented the capacity of the palladium or the capacity of the silver.

Polarization characteristics were about normal for a hydrogen electrode catalyzed with pure palladium. No reliable results were obtained for individual electrodes as the hydrogen reference electrode failed repeatedly, probably because it was a platinum catalyzed, sintered nickel electrode and a hydrogen gas pressure could not be maintained on it in the presence of the sintered palladium electrode.

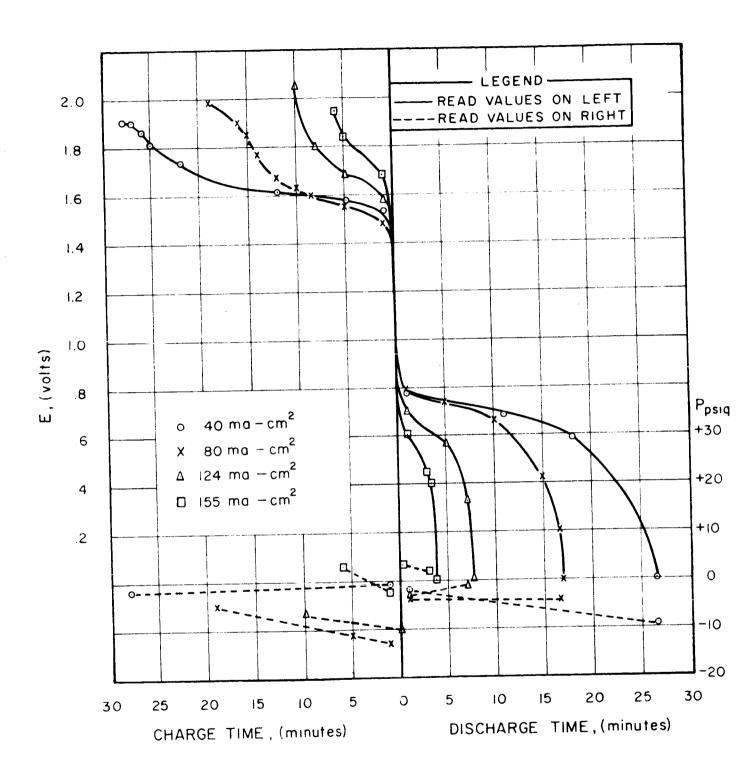


FIG. 14 $A_{A_2}^{0}$ VS $Pd(H_2)$

6. FUTURE PLANS

The electrolyte storage experiments will be continued through January, and the charge retention studies will be run concurrently. The first experiments are being conducted at 20 ma/cm², and further experiments on electrolyte storage will be done at higher current densities. If the cell appears to be limited to rather low current densities in these experiments, then higher temperature experiments will be performed.

Some experiments to determine cycle efficiencies will complete the program. A few additional experiments, however, will be conducted to finish the work of the first phase. Some studies of hydrogen storage in Raney nickel will be made and a redetermination of KOH concentration dependence on electrode polarization will be made.

7. APPROXIMATE MANHOURS AND DOLLAR EXPENDITURES AND COMMITMENTS FOR PERIOD 25 SEPTEMBER - 25 DECEMBER 1962:

MANHOURS

J. Rowlette, Project Supervisor 362 hours D. Lee, Chemist 508 hours E. Avis, Research Chemist 148 hours

DOLLAR EXPENDITURES AND COMMITMENTS: \$12,912

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- 3. T. P. Dirkse, "The Oxidation of the Silver Oxide Electrode in Alkaline Solution", J. Electrochem. Soc., <u>106</u>, 920-5 (1959)